Organic Compounds - HPLC

Assessor Checklist

Assessor's Name _____ **Date** _____

Laboratory _____

Location _____

Item	Section 1 - Personnel	Reference	Y	N	NA	Comments
1.0	Does the analyst(s) interviewed have the necessary education, training, technical knowledge and experience for their assigned functions?	NELAC - 5.6.1 and 3.5.3.d				
	A. Initial demonstration of capability?	NELAC - 5.6.2.b & 5C				
	B. Analyst's file contain a certificate that they have read, understood and agreed to perform most recent version of the test method?	NELAC - 5.6.2.c.3				
	C. Documentation of continued proficiency at least once per year?	NELAC - 5.6.2.c.3				

	Section 2 - Sample Receipt/Preservation	Reference	Y	N	NA	Comments
2.0	Are samples kept at 4°C during transport?	NELAC - 5.11.3.a.1				
2.1	Are samples preserved?	NELAC - 5.11.3.a.2				
	A. pH 3.0?[1]	531.1, 6610B				
	B. ≤ pH 2.0?	550.1, 555				
	C. Sodium thiosulfate if residual chlorine?	531.1, 6610B, 547, 549.1				

[1]All method specific criteria in this check sheet only apply when a mandated method is being reviewed.

2.2	Are samples stored correctly? A. At -10°C?	NELAC - 5.11.4.a 531.1 and 6610B				
2.3	Is the HPLC organic analysis laboratory(s) and the sample storage area(s) separated from incompatible areas so that contamination does not affect data quality?	NELAC - 5.7.2, 5.11.4 & 5.11.4.a.2				
2.4	Is the sample bottle checked for damage or leaking prior to analysis?	NELAC - 5.11.2.e & f				
2.5	Are samples analyzed within the appropriate hold time specified in the 40 CFR? A. If not, is the report qualified?	NELAC - 5.11.2.d and 5.11.3.c NELAC - 5.11.2 and 5.11.3.c				

	Section 3 - Equipment	**Reference**	**Y**	**N**	**NA**	**Comments**
3.0	Does the laboratory have all the HPLC equipment required for the correct performance of tests for which accreditation is sought?	NELAC - 5.8a				
3.1	Is the HPLC equipment properly labeled, maintained, inspected, and cleaned with documentation?	NELAC - 5.8.b, d and e				
3.2	Is equipment that is not operating properly taken out of service?	NELAC - 5.8.c				

	Section 4 - Method and Essential Quality Control Requirements		**Y**	**N**	**NA**	

		Reference				Comments
4.0	Does the HPLC laboratory section have approved SOPs for each parameter that accreditation is sought? A. Accessible to all personnel? B. Specific SOP references: _____ _____ _____	NELAC - 3.5.3.f NELAC - 5.10.1.1.c &e				
4.1	Do the HPLC SOPs accurately reflect the actual activities of the section, including analysis of samples and standards under identical conditions?	NELAC - 5.10.1.1				
4.2	Has the laboratory maintained on record all original observations, calculations and derived data, calibration records and a copy of the test report?	NELAC - 5.12				
4.3	Can these records be historically reconstructed of all laboratory activities that produces the resultant sample analytical data?	NELAC - 5.12.1				
4.4	Are these records available for the past 5 years, if applicable?	NELAC - 4.3.3				
4.5	Are records entries or changes made according to the NELAC Standard?	NELAC - 5.12.1.d, e and f				
4.6	Is computer electronic data maintained so that the integrity of the calibration and test data can be reviewed?	NELAC - 5.10.6				
4.7	Has the assessor verified the data quality systems and essential quality control requirements of Chapt. 5 by reviewing random representative data packets from the HPLC organic laboratory section, including COC records; raw data, including hard copy and electronic data; and all further records that lead to the analyte results recorded on the final report? (Note: Raw data review should include making sure that chromatographic analyte peaks are appropriately integrated (by the computer or manual) to be consistent (unless effected by matrix) and scientifically defensible. Individual peaks on hard copy data may need to be blown up by the software to adequately ascertain if a peak is integrated appropriately)	NELAC - 3.5.3.g, j, k,l, and 3.6.4.h				
4.8	Are standards prepared, handled, labeled, and stored per defined procedures and does it include a program to use verified standards and replace as required?	NELAC - 5.10.1.1, 5.8.d, 5.9.2, 5.9.3, 5.9.4 and 3.5.3.h and l				

4.9	Is if, over the calibration range, the RSD of response factors is less than 15 percent, or the RSD of calibration factors is less than 30 percent, linearity through the origin can be assumed and an average relative response factor bay be used; other wise the complete calibration curve shall be used?	NELAC - 5.9.4.3.c.1				
4.10	Is if a linear regression is used, the correlation coefficient (R) shall be no less than 0.995 unless the laboratory can demonstrate that a lowered correlation coefficient consistently produces accurate results?	NELAC - 5.9.4.3.c.2				
4.11	<u>Mandated HPLC Analyses' Calibration</u> (5.10.2.a1) A. Are a minimum of three standards used to calibrate? B. Is calibration for each analyte performed with the lowest standard near but above the respective EDLs, and with the remaining standards concentrations that bracket the working range? C. Is the average calibration factor or response factor used when the RSD is less than (X) for the calibration range of standards? 1. X = 10%? 2. X = 20%? D. Is the single calibration point within ±20% of the sample concentration when using the single point method?	 531.1, 6020B, 547, 6651, 549.1, 550.1, and 555 531.1, 6610B, and above MDLs for 547, 550.1 & 555 547, 550.1 531.1, 6610B, 6651, 555 531.1				
4.12	Is when an initial calibration curve is not established on the day on analysis, the integrity of the initial calibration curve verified on each day of use (or 24 hours period) by initially analyzing a blank and a standard at the method defined concentration or a mid-level concentration if not included in the method? A. Initial calibration verification within 15% of the true value unless the lab can demonstrate through historical data that wider limits are applicable? B. Analysis procedure stopped and evaluated after initial calibration verification fails? C. Analysis only begins once initial calibration verification acceptable?	NELAC - 5.9.4.4.1.a NELAC - 5.9.4.4 NELAC - 5.9.4.4.1.b NELAC - 5.9.4.4.1.b				

4.13	Are continuing calibration verification standards analyzed after the initial calibration curve or the integrity of the initial calibration curve has been accepted?	NELAC - 5.9.4.4.2				
	A. 15 % of true value, unless lab proven ?	NELAC-5.9.4.4				
	B. Mandated Methods:					
	1. 20% of the predicted response?	531.1, 547, 549.1, 550.1 555				
	2. 25% of predicted response?					
	C. Analyzed at a frequency of 5%or every 12 hours whichever is more frequent?	NELAC - 5.9.4.4.2.a				
	D. Concentration determined by the anticipated or known concentration of the samples and /or method specific levels with one being a low level concentration?	NELAC - 5.9.4.4.2.b				
	E. New calibration curve run, if calibration check standard fails and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria?	NELAC - 5.9.4.4.2.c				
	F. Sample results only reported if CCV results high and there are non-detects for the corresponding analyte in all samples associated with the CCV?	NELAC - 5.9.4.4.2.c				
4.14	Is a method blank carried through all stages of the sample preparation and measurement run at a frequency of one per batch of samples per matrix type per sample extraction or preparation method??	NELAC -5D.1.1.a.1				
	A. Corrective action taken when blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch?	NELAC -5D.1.1.a.1.i				
	B. Corrective action taken when the blank concentration exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit?	NELAC - 5D.1.1.a.1.ii				
	C. Each sample affected by either a or b above assessed and reprocessed or reported with appropriate data qualifier?	NELAC - 5D.1.1.a				

4.15	Is a laboratory control sample analyzed at a minimum of 1 per batch of 20 or less samples per matrix per sample extraction or preparation method and the results used to determine batch acceptance? A. All reported components spiked unless method specifies otherwise?	NELAC - 5D.1.1.b.1 NELAC - 5D.1.1.b.4				
4.16	Are matrix spikes performed at frequency of one in 20 samples per matrix type per sample extraction or preparation method and poor performance reported to the client? A. All reported components spiked unless method specifies otherwise?	NELAC -5D.1.1.b.2 NELAC - 5D.1.1.b.4				
4.17	Are matrix spike duplicates performed at a minimum of 1 in 20 samples per matrix per sample extraction or preparation method and poor performance reported to the client? A. All reported components spiked unless method specifies otherwise?	NELAC - 5D.1.2 NELAC - 5D.1.1.b.4				
4.18	Are surrogate compounds added to all samples, standards, and blanks for all volatile chromatography methods except when the matrix precludes its use or when a surrogate is not available and poor results reported to client? A. All reported components spiked unless method specifies otherwise?	NELAC - 5D.1.1.b.3 NELAC - 5D.1.1.b.4				
4.19	Has the laboratory developed and documented acceptance criteria for retention time windows?	NELAC - 5D.1.7.a				
4.20	Do samples with positive detected results have their compound identification confirmed (except for HPLC/MS analyses)?	NELAC - 5D.1.7.b				
4.21	Are results to be reported as quantitative bracketed by calibration or calibration verification standards and all other results reported as having a lower confidence level?	NELAC - 5.9.4.3.d				
4.22	Do all reports sent to an outside client have data qualifiers when there are deviations from (such as failed quality control), additions to or exclusions from the test method (Such as environmental conditions), and any non-standard conditions that may have affected the quality of results? A. Are all other report contents required by NELAC present?	NELAC - 5.13.a.10 NELAC - 5.13				

References:

-NELAC Standards - Approved July 1998, EPA600/R-98/151, November 1998, www.epa.gov.

-EPA Method 531.1, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA-600/4-88-039, December 1988, Revised, July 1991.

-EPA Methods 547 and 550.1, *Methods for the Determination of Organic Compounds in Drinking Water-supplement I*, EPA-600-4-90-020, July 1990.

-EPA Methods 549.1 and 555, *Methods for the Determination of Organic Compounds in Drinking Water-Supplement II*, EPA-600/R-92-129, August 1992.

-Methods 6020B, 6610B and 6651, *Standard Methods for the Examination of Water and Wastewater*, 18th edition, 1992, APHA.

Assessor's Notes: